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Progress Report on U.S. Research on Test Methods and Materials

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1. TEST METHODS

In the United States, the most important fire standards writing organization is the American Society for Testing and Materials (ASTM). Fire standards are also, however, developed by the National Fire Protection Association (NFPA), and Underwriters Laboratories (UL), but they are often similar to the ones issued by ASTM.

ASTM has issued over 100 standards associated with various aspects of fire, including guides, terminologies and specifications as well as test methods. Within ASTM, committee E5 is specifically dedicated to writing generic fire standards. However, many other committees, which have different primary responsibilities, also write fire standards which are specific to the materials, products or occupancies of their concern. For example, committees D7 (on Wood), D9 (on Electrical Insulation Materials), D11 (on Rubber), D13 (on Textiles) and D 20 (on Plastics) all write fire test methods for their materials, as do committees F15 (Consumer Products) and F23 (Protective Clothing). Committees interested in Aircraft (F7), Ships (F25) or Correctional Facilities (F33), also write test methods that deal with fire issues. The process by which ASTM develops consensus test methods is public, and every concern expressed must be addressed, through a relatively strenuous procedure.

At NFPA, all fire tests are developed by the Fire Test committee, the members of which are appointed, and the results of whose deliberations are published. Comments can then be made by the public and the final recommendations are voted on in public meetings by the entire membership. Such standards generally tend to contain pass-fail criteria, while those of ASTM tend to be generic. UL standards are not generated by public consensus. UL is a private organization which canvasses industrial opinions, and makes its own decisions.

All of these organizations create various types of fire standards, mainly guidance documents and test methods addressing most of the major fire properties. Only newly adopted test methods during the last several years are described here.

SMOKE OBSCURATION

ASTM E662, often known as the NBS smoke density chamber, is extensively referred to in specifications and requirements, and quoted in published data. However, this test method has several limitations such as the use of white light (uncertainty over integrating over a broad emission spectrum), vertical sample mounting configuration (dripping problem for certain materials), change in oxygen concentration in the test chamber during an experiment, deposition of soot and condensable species on walls and optical components, and others. A modification of this test method has been drafted in ASTM (draft ASTM E5.21.3) and has also been standardized internationally (ISO 5659, Part 2), which differs from the original in that the heat source is a conical radiant heater, similar (but not identical) to the cone heater in ASTM E1354 (Cone Calorimeter Test), piloted ignition is achieved by means of a spark igniter, the specimen is oriented horizontally, and there is an optional capability for a load cell, which measures sample mass loss continuously during a test. The incident heat flux can be set at any value, but values of 25 and 50 kW/m^2 are required in the standard. The repeatability and reproducibility of this test method are better than those of ASTM E662, and it also solves some of the limitations of that procedure.

HEAT RELEASE RATE

Bench-scale:

ASTM E1474 (or NFPA 264A) is an application of the Cone Calorimeter (ASTM E1354) specifically for use with upholstered furniture or mattress composite specimens. It determines the same flammability characteristics as ASTM E1354, but specifies a particular incident heat flux of 35 kW/m^2, and a specific specimen preparation and mounting procedure [1]. In fact, the standard allows two specimen preparation procedures, with one of them suggested for screening purposes only. The method deemed acceptable for final testing was developed as a result of work funded by the European Commission, and conducted by a testing conglomerate under the designation "Combustion Behavior of Upholstered Furniture" [2]. ASTM F1550 is based on ASTM E1474, for direct applicability to correction and detention facilities. It addresses the testing of upholstered furniture or mattress composite specimens, but in a vandalized fashion, by slashing through the fabric and any potential interliner present. The objective of this test method is to prevent the use, in correction and detention facilities, of paddings posing excessively high fuel loads. Such paddings may be simply protected by a pierceable barrier, so that they do not ignite easily under normal circumstances, but cause a severe fire hazard when they do eventually burn. Other applications of the Cone Calorimeter also exist: ASTM E1740 was approved in 1995, for use with wall covering composites (including the backing material) and a new standard is being drafted for use with electrical wires and cables and their coating materials.

Real-scale:

ASTM E1537 (or NFPA 266), ASTM E1590 (or NFPA 267) and a draft test for stacking chairs represent a new generation of fire tests: a real-scale item (either an upholstered furniture piece, a mattress, or a stack of chairs) is placed on a load cell in a furniture calorimeter, or in a room, and ignited by a gas burner that is on for a fixed period of time. The release rates of heat, smoke, and combustion products are determined by measurements in the exhaust duct. It has been shown that, for peak heat release rates of less than 600 kW, heat release is not affected by the re-radiation from the walls, so that testing in a small or in an open furniture calorimeter should give similar results [3]. Therefore, ASTM E1537 involves upholstered furniture, which can be tested under the hood in a furniture calorimeter or inside a small room, either of dimensions 2.4 by 3.7 m or 3.0 by 3.7 m, with a 2.4m height and a standard door opening. The ignition burner is square shaped, and the flame is turned on for 80 s, at a propane flow rate of 13 l/min. The application for this test method is contract occupancies, of higher than average risk, particularly in the absence of sprinklers. The test is based on the concepts put forward in California Technical Bulletin TB 133 and contains a set of pass/fail criteria, based on heat release: 80 kW peak rate of heat release and 25 MJ total heat released in the first 10 min. of test. NFPA 266 is equivalent to ASTM E1537, except that testing must be conducted in a furniture calorimeter. This test method has been adopted for regulation in some states, and has been incorporated into the generic sections of the National Life Safety Code, NFPA 101, as well as into the specific sections related to detention and correction occupancies. The requirements set are a maximum rate of heat release of 250 kW and total heat release of no more than 40 MJ in the first 5 min of test. Similarly, ASTM E1590 has also been adopted by various states and by NFPA 101.

Room-corner tests: The heat release rate of wall lining materials is determined in full scale, by oxygen consumption calorimetry, by lining walls (or walls and ceiling) of a small room (2.4 by 3.7 m by 2.4 m high) with the products under consideration. In ASTM E-5.13.2 Draft, wall lining materials lining three walls are tested (all the walls except the one containing the door opening); it utilizes a gas burner, placed flush against walls in the corner at a height of 305 mm from the floor. The burner is set at an incident power of 40 kW for 5 minutes, followed by a setting of 160 kW for a further 10 min. The measurements, all made in the exhaust duct, include heat and smoke release, but the principal decision to be made is whether the wall lining is able to prevent the flames from reaching the outer extremities of the test specimen and the room from reaching flashover.

Large Scale Tests for Cables: ASTM has issued two vertical cable tray tests for cables to be used in industrial facilities. They are ASTM D5424 and ASTM D5537 and they measure smoke release (D5424) and heat release (D5537) together with flame spread and mass loss on 2.4 m lengths of cables.

Smoke Toxicity

Both ASTM and NFPA have been working for many years in developing a standard test for toxic potency of smoke. ASTM has recently issued ASTM E1678, a test method for determining toxic potency. The apparatus consists of a 200 L plastic exposure chamber communicating, through a connecting chimney, with a combustion chamber. Sample combustion results from radiant exposure to a flux of 50 kW/m^2, generated by a set of quartz lamps, for 15 min. Concentrations of the major gaseous toxicants are monitored over a 30 min period, with concentration-time products for each being determined from integration of the areas under the respective concentration-time plots. They are then used to determine the preliminary analytical toxic potency (LC_{50}) by using N-gas model equations, where toxic potency is calculated as an additive function of the toxic potencies of the individual gases. Unfortunately, the standard only addresses pre-flashover fire situations, and toxic potency values for many products are separated only by less than a factor of three. Work is in progress to develop an alternative standard, or changes to E1678, to address toxic potency measurements for post-flashover scenarios, with the same test apparatus (by requiring correction for the carbon monoxide concentration inevitably present in post-flashover scenarios). NFPA 269 includes both the test method and the corrections needed for post-flashover scenarios.

Smoke Corrosivity

ASTM has issued the cone corrosimeter (ASTM D5485). In it the cone calorimeter is used to burn small samples of materials or products, and an aliquot of the resulting smoke is passed through an exposure chamber where copper circuit boards are exposed for 1 hour. The targets are then removed from the exposure chamber and kept for 24 hours post-exposure, at room temperature and 75%

relative humidity. Another test method is in the process of being developed, using the same test apparatus as the smoke toxicity standard E1678, and the same corrosion targets and post-exposure protocol as ASTM D5485.

2. Fire Safe Materials

A new major research thrust has been put forward for the development of advanced fire safe materials applicable to a commercial aircraft interior by the Technical Center of the Federal Aviation Agency, FAA. The National Research Council has recently published a report describing recommendations on this matter [4]. The research activity at this center on this topic will be presented by Dr. Lyon. Many universities are participating in this thrust and their projects are briefly described below.

• Case Western Reserve University: Synthesis and characterization of polybenzoxazines - a new thermally-stable, high char yield polymer. The polybenzoxazines are superior in thermal stability and comparable in price and ease of processing to phenolic resins without the drawbacks of free formaldehyde or gaseous product generation during curing.

• Cornell University: Thermal stability enhancement by molecular-level reinforcement and its relationship to flammability will be studied for engineering polymer nanocomposites.

• Richard Stockton State College of New Jersey: Synthesis of new cyanate ester monomers leading to addition-cured triazine resins for interior panels and adhesives with high thermal stability, low heat of combustion, and low combustion efficiency will be conducted.

• Rutgers University: Mechanical properties and fracture behavior of carbon-fiber reinforced polysialate primary and secondary aircraft composites will be studied at ambient and elevated temperatures and related to constituent material interactions and process variables.

• Pennsylvania State University: Linear polychlorophosphazenes using an economical synthetic route will be synthesized and evaluated for miscibility and fire resistance in blends and composites.

• University of Massachusetts: Molecular design of fire safe polymers and composites for various interior applications will be conducted using computational and synthetic chemistries. Mechanistic approaches to improve fire resistance include thermodynamic phase changes and chemical reactions during fire exposure which consume heat, generate flame suppressants, or increase char formation.

• University of Michigan: A family of inorganic-organic polymer will be synthesized for various aircraft applications requiring thermally stable resins, low viscosity liquid crystalline materials, high silicon-content elastomers, and low density thermal insulation.

• University of Akron: Molecular dynamic simulations and experimental kinetic studies of polymer pyrolysis in the bulk, at surfaces, and with coupled diffusion of small molecules (degradation products, flame retardants, oxygen) will help researchers to design materials which will provide a totally fire resistant aircraft cabin.

• University of South Carolina: A new class of fire safe polymeric materials containing no halogens or heteroatoms will be synthesized; it is based on soluble and processable oligomeric and polymeric alkyne-functionalized polyphenylenes and fullerenes.

• Virginia Polytechnic Institute and State University: Synthesis, characterization, and fire evaluation of phosphine oxide copolymers of polyetheretherketone, polyetherketone, polypheneylene sulfide, and polyimide.

At NIST, new non-halogenated flame retardant approaches are being explored mainly for large volume commodity polymers such as polyethylene, polypropylene, Nylon, polystyrene, and others. One approach is to enhance char formation by an appropriate combination of additives to these polymers. Reduction in heat release rate by forming char has been demonstrated by the use of potassium carbonate and silica gel combination in these polymers [5]. Another approach using silicon compounds is also being explored. The molecular dynamic model to calculate thermal degradation behavior of polymers has been further extended by combining its code with the DISCOVER code of Biosym/Molecular Simulations. This extension allows it to apply to many different polymers[6]. A new model is being developed to describe physical and chemical processes yielding an intumescent char. The model is numerically solved for the time-dependent behavior of growth and transport of numerous bubbles in a polymer slab and subsequent swelling of the polymer sample. With the addition of carbonization chemistry and its physical effect, the model can be used to explore how to design intumescent materials at the optimum condition to reduce gasification rate/heat release rate most effectively [7].

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